



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Suresh Shankarappa VAGARALI *et al.*

Application No. 09/162,206

Group Art Unit: 1754

Filed: September 28, 1998

Examiner: S. Hendrickson

For: HIGH PRESSURE/HIGH TEMPERATURE
 PRODUCTION OF COLORLESS AND
 FANCY COLORED DIAMONDS

DECLARATION UNDER 37 CFR § 1.132

I, Suresh Vagarali, do hereby declare to my own knowledge that:

1. I am currently employed as a Technology Development Leader at GE Superabrasives, an affiliate company of GE Superabrasives of Ireland, an assignee of the above identified application.
2. I received my Ph.D. in Materials Science from University of Southern California in 1979.
3. I have over 14 years experience in superabrasive materials, synthetic diamonds, and natural diamonds, including processing of natural diamonds and synthesis of gem quality jadeite.
4. I am an inventor or co-inventor of five U.S. patents. I have sixteen publications in technical journals. My CV is attached as Exhibit A.
5. I make this declaration to provide data in support of the above identified patent application.
6. I have prepared under my direct supervision and control the following experiments to evaluate the effect of pressure and temperature for changing the color of diamonds as disclosed in U.S. Patent No. 3,134,739 ("Cannon"). These experiments were run to determine the veracity of the Cannon invention as defined by the statement, "At high pressures and temperatures, diamonds grown become more clear and white, but aluminum diffusion provides a more marked and contrasting change to colors." (Cannon, column 6 line 73 to column 7, line 1). Experiments 2-4 below repeat Examples 5, 4 and 6, respectively, of Cannon, column 4, lines 48-60. Experiment 1 below repeats Example 5 of Cannon except that aluminum disks are not used.

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7. All Experiments were run using Type Ia natural brown diamonds, which falls within Cannon's disclosure referencing natural and man-produced diamonds generally. See Cannon, col. 3, lines 16-20. The Examples in the specification demonstrate the claimed methods using Type Ia natural brown diamonds (Examples III and IV) and Type IIa natural brown diamonds (Examples I, II, V and VI). The claims of this application are currently directed towards methods for changing the color of colored Type II natural diamonds.

Experiment 1. A brown Type Ia natural diamond weighing 0.1957 gm was embedded in 0.500" thick graphite pill. The graphite used for pressing the pill is Carbone grade MF2027P powder with maximum impurity content of 10 ppm. The pill was placed inside MgO cups and then in graphite heater tube. The cell assembly was subjected to 60 kbar¹ pressure, 1500° C temperature for 20 minutes. After the completion of the run, the diamond was removed from the cell and was cleaned using molten salt bath containing 96% NaOH and 4% KNO₃ at 1250° F for 25 minutes.

Experiment 2. A brown Type Ia natural diamond weighing 0.2335 gm was embedded in 0.330" thick graphite pill made of Carbone MF2027P powder. Four aluminum discs each with thickness of 0.010" were placed on both side of the graphite pill. The aluminum content of the discs was 99.4% (minimum). Finally, 0.045" thick graphite pills were placed at the ends bringing the total thickness of the assembly to 0.500". The above assembly was placed inside MgO cups and then in graphite heater tube. The cell was subjected to 60 kbar, 1500° C temperature for 20 minutes. After completion of the run, the diamond was removed from the cell and was cleaned by using hot acid mixture of 90% sulfuric acid and 10% nitric acid for 2 hours. After acid cleaning the diamond was rinsed using distilled water.

Experiment 3. A brown Type Ia natural diamond weighing 0.2557 gm was embedded in 0.330" graphite pill. The remaining details of the cell assembly are the same as in Experiment 2. The cell was subjected to 35 kbar pressure and 1100° C for 20 minutes. The diamond was recovered from the cell and then cleaned using the same acid cleaning procedure as in Experiment 2.

Experiment 4. A brown Type Ia natural diamond weighing 0.1957 gm (same as in Experiment 1) was embedded in 0.330" thick graphite pill. The remaining details of the cell assembly are the same as in Experiment 2. The cell was subjected to 60 kbar pressure and 1400° C temperature for 20 minutes. The diamond was recovered from the cell and then cleaned using the same acid cleaning procedure as in Experiment 2.

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¹ 1 bar of pressure equals approximately 1 atmosphere of pressure, so 60 kbar equals approximately 60,000 atm.

The color / appearance of each diamond before and after the high pressure / high temperature treatment in the reactor vessel was recorded. The results are shown below:

Example	BEFORE color of diamond	Reactor Pressure (kbar)	Reactor Temperature °C	Time (minutes) in Reactor	Use of Al. disks	AFTER color of diamond	OBSERVED change in diamond color
1	Brown	60	1500	20	N	Brown	None
2	Brown	60	1500	20	Y	Brown	None
3	Brown	35	1100	20	Y	Brown	None
4	Brown	60	1400	20	Y	Brown	None

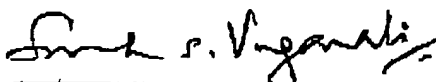
8. The diamonds were examined BEFORE and AFTER the experiments. They remained the same brown color. I did not see any change in color in any of the four experiments. I therefore conclude that the statement in Cannon that "diamonds grown become more clear and white, but aluminum diffusion provides a more marked and contrasting change to colors," is false (Cannon, column 6 line 74 to column 7, line 1). I further conclude that no observable change in color occurs in diamonds that are processed under the pressure and temperature conditions disclosed in Cannon.

9. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

August 13, 2002

Date



Suresh S. Vagarali

Exhibit A: CV of Suresh S. Vagarali

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EXHIBIT A: CV of Suresh S. Vagarali**Education:**

Ph. D. Materials Science, 1979, University of Southern California, Los Angeles, CA.
B. S. Metallurgical Engineering, 1969, University of Mysore, Mysore, India

Training:

Robust Design (2001)
DFSS Training (2000)
Leadership Skills Development Course (1998)
Six Sigma (MAIC) Training (1996)

Employment History:

7/1997 to Present: Technology Development Leader, GE Superabrasives, Worthington, OH.
7/1988 to 6/1997: Sr. Research Engineer, GE Superabrasives, Worthington, OH.
2/1988 to 6/1988: Sr. Research Engineer, Norton Company, Troy, NY.
4/1982 to 2/1988: Sr. Research Engineer, Norton Company, Worcester, MA.
1/1979 to 4/1982: Asst. Research Engineer, University of California, Santa Barbara, CA.

Qualifications:

- Lead inventor of patent on Synthetic Imperial Grade Jadeite, approval received from patent office (2001)
- Winner of 1999 GE Plastics CHARLES E. REED Process Innovation Award for work on Bellataire Gem Diamond Project (2000)
- Award of Stock Options and Restricted Stock Units for work on Bellataire Gem Diamond Project (1998)
- Five US/European Patents received and seven applications pending
- Strong technical background in materials research (cell design and synthesis of new products)
- Expertise in transfer of technology to an overseas plant
- Three management awards at GE Superabrasives
- Sixteen publications in technical journals

List of U.S. Patents:

6,377,340: Method of detection of natural diamonds that have been processed at high pressure and high temperatures
5,869,015: Method for producing cubic boron nitride using melamine as catalyst
5,022,894: Diamond compacts for rock drilling and machining
4,741,743: Grinding wheel with combination of fused and sintered abrasive grits
4,609,381: Grinding aid

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